

**AD-A234 315**

## REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS		
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release distribution unlimited.		
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE			5. MONITORING ORGANIZATION REPORT NUMBER(S)		
4. PERFORMING ORGANIZATION REPORT NUMBER(S)			7a. NAME OF MONITORING ORGANIZATION ONR		
6a. NAME OF PERFORMING ORGANIZATION Kensselaer Polytechnic Institute		6b. OFFICE SYMBOL (if applicable)	7b. ADDRESS (City, State, and ZIP Code) Department of Navy Arlington, VA 22217		
6c. ADDRESS (City, State, and ZIP Code) Department of Chemistry Troy, New York 12180-3590		8b. OFFICE SYMBOL (if applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER Contract # N00014-85-K-0632		
8a. NAME OF FUNDING/SPONSORING ORGANIZATION ONR		10. SOURCE OF FUNDING NUMBERS			
8c. ADDRESS (City, State, and ZIP Code) Department of Navy Arlington, VA 22217		PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) A Program in the Chemistry of Electronic Materials. Task I. New Organometallic Precursors for the Low Pressure Chemical Vapor Deposition of Refractory Materials for Electronics					
12. PERSONAL AUTHOR(S) L.V. Interrante.					
13a. TYPE OF REPORT Final Report		13b. TIME COVERED FROM 7/1/85 TO 12/31/90		14. DATE OF REPORT (Year, Month, Day) 28 February 1991	
				15. PAGE COUNT 11	
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP	Chemical vapor deposition, thin films, AlN, SiC, Si <sub>3</sub> N <sub>4</sub> , organometallic precursors		
19. ABSTRACT (Continue on reverse if necessary and identify by block number)  Please see Abstract on the reverse side					
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION Unclassified		
22a. NAME OF RESPONSIBLE INDIVIDUAL Dr. Mark Ross			22b. TELEPHONE (Include Area Code) (202) 696-4409		22c. OFFICE SYMBOL

91 4 03 024

# 19. ABSTRACT

The principal focus of this Project was the design and synthesis of volatile organometallic precursors for the LPCVD of AlN, SiC, and other materials of interest for applications in electronics. As potential precursors for AlN, and SiC, various cyclic compounds of the type,  $[A(XX')B(Y Y')]_n$  (where A, B = Al, N; or Si, C; X, X', Y, Y' = H, CH<sub>3</sub>, etc. and n = 2 and 3), were prepared and studied. The pyrolytic decomposition of these compounds were investigated using mass spectrometry and IR spectroscopy and the resultant thin films examined by Auger spectroscopy and SEM/TEM to determine the composition, microstructure, and purity of the phases present. Through the use of selected precursors of this type, high quality, films of stoichiometric AlN and SiC were obtained at temperatures (500 - 800 °C) well below that employed in previous investigations.

Thin films of both AlN and the cuprate superconductors were also prepared by pyrolysis of solution-applied polymeric precursor films. The AlN films were obtained from ethylenediamine:triethylaluminum derivatives and the cuprate superconductors from citric acid/ethyleneglycol mixtures containing the respective metal nitrate salts. The structures and pyrolysis reactions of these polymeric precursors were studied by NMR, FTIR, XRD and other methods.

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OFFICE OF NAVAL RESEARCH

FINAL REPORT

for

Contract N00014-85-K-0632

R&T Code 413c014

A Program in the Chemistry of Electronic Materials  
At the Department of Chemistry  
Rensselaer Polytechnic Institute

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28 February 1991

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## TASK I

### NEW ORGANOMETALLIC PRECURSORS FOR THE LOW PRESSURE CHEMICAL VAPOR DEPOSITION OF REFRACTORY MATERIALS FOR ELECTRONICS

Prof. L.V. Interrante

#### SUMMARY

The principal focus of this Project was the design and synthesis of volatile organometallic precursors for the LPCVD of AlN, SiC, and other materials of interest for applications in electronics. As potential precursors for AlN, and SiC, various cyclic compounds of the type,  $[A(XX')B(YY')]_n$  (where A, B = Al, N; or Si, C; X,X',Y,Y' = H, CH<sub>3</sub>, etc. and n = 2 and 3), were prepared and studied. The pyrolytic decomposition of these compounds were investigated using mass spectrometry and IR spectroscopy and the resultant thin films examined by Auger spectroscopy and SEM/TEM to determine the composition, microstructure, and purity of the phases present. Through the use of selected precursors of this type, high quality, films of stoichiometric AlN and SiC were obtained at temperatures (500 - 800 °C) well below that employed in previous investigations.

Thin films of both AlN and the cuprate superconductors were also prepared by pyrolysis of solution-applied polymeric precursor films. The AlN films were obtained from ethylenediamine:triethylaluminum derivatives and the cuprate superconductors from citric acid/ethyleneglycol mixtures containing the respective metal nitrate salts. The structures and pyrolysis reactions of these polymeric precursors were studied by NMR, FTIR, XRD and other methods.

#### BACKGROUND

The present study was begun in 1985 in the context of a multi-investigator program in the chemistry department at Rensselaer that was directed broadly at the application of chemistry to electronics. This program seeded the development of three different research efforts in electronic materials chemistry and made possible the purchase of a TGA/DSC

system which we have used extensively in the course of our precursor synthesis and evaluation efforts.

The program also provided funds for the construction of a cold-wall, hot-stage CVD system in our laboratory which has been used to evaluate new CVD precursors and to prepare thin films of AlN and SiC for examination of electrical and optical properties. This apparatus has recently been upgraded by adding a mass flow controller and quadrupole mass spectrometer, allowing control of precursor flow rates and on-line mass spectral analysis of precursor decomposition products. A more detailed description of this apparatus is included as Appendix A of this report.

## RESULTS

A major focus of this program has been the design and synthesis of volatile organometallic precursors for the low pressure chemical vapor deposition of aluminum nitride and silicon carbide for applications in electronics. As potential precursors for these materials, various cyclic compounds of the type,  $[A(XX')B(Y Y')]_n$  (where A, B = Al, N; or Si, C; X, X', Y, Y' = H, CH<sub>3</sub>, etc. and n = 2 and 3), were prepared and studied. In addition, a monomeric silicon tetraamide was examined as potential low pressure CVD precursor to Si<sub>3</sub>N<sub>4</sub>. The study of these compounds has included the investigation of their pyrolytic decomposition, by analysis of the volatile decomposition products, and also the examination of the product thin films to determine their composition, microstructure, purity and properties.

The cyclic precursor,  $[Me_2AlNH_2]_3$ , was used to deposit high quality, polycrystalline thin films of AlN on Si and other substrates at considerably lower temperatures than had been required previously to obtain films of this type. Separate studies of this precursor system in solution, by NMR spectroscopy, revealed a rapid equilibrium involving the dimeric and, presumably, the monomeric form of this compound and Generalized Valence Bond calculations were performed that indicated a planar structure and a significant stability for this monomer. Recent mass spectral studies carried out in collaboration with J. Hudson have suggested that this trimer-dimer equilibrium extends also to the gas phase and that the active species in the CVD process may be one of these other forms of the  $[Me_2AlNH_2]_3$  precursor. Efforts to study both the gas phase and surface

chemistry of this precursor are continuing.

In the course of this program a new volatile precursor to SiC was also identified and used to prepare thin films of apparently stoichiometric SiC at temperatures as low as 600 °C. This precursor is a volatile, liquid, cyclic carbosilane,  $[\text{MeSi}(\text{H})\mu(\text{CH}_2)_2\text{Si}(\text{Me})\text{CH}_2\text{SiH}_2\text{Me}]_2$ , derived from the Grignard coupling of  $\text{Cl}_2\text{Si}(\text{Me})\text{CH}_2\text{Cl}$ . It was employed as a single-component precursor for SiC deposition on a Si(100) surface in both a hot-wall,  $\text{SiO}_2$  reactor and in Prof. Hudson's ultrahigh vacuum system. In both cases smooth, dense, adherent SiC films were obtained; however, the microstructure of the deposit was found to depend on the reaction conditions, with a fine-grained amorphous deposit resulting from the hot-wall reactor at 600-800 °C and a polycrystalline deposit from the experiments in the ultrahigh vacuum system. Thermal desorption experiments carried out in Professor Hudson's laboratory have revealed a two-peak desorption profile which appears to relate to the cis- trans-isomer distribution present in the precursor source. The onset of a chemical process leading to CVD of stoichiometric SiC at ca. 500 °C was also evidenced.

Efforts to deposit  $\text{Si}_3\text{N}_4$  using volatile, single-component precursors centered on the use of silazanes in which the Si is bound only to nitrogen. Initial work with  $\text{Si}(\text{NHEt})_4$  gave black films on Si which contained Si, C, and N by Auger analysis. The proportion of N to C was found to depend on the deposition conditions; however, significant amounts of carbon were observed in all cases. Preliminary results suggested that a significant reduction in the carbon content could be achieved by the addition of ammonia to the gas stream containing this precursor. Subsequently, this conclusion was verified in an independent study by Gordon and Hoffman who used this precursor along with  $\text{NH}_3$  in an atmospheric pressure CVD experiment to obtain an amorphous  $\text{Si}_3\text{N}_4$  film at 600 °C.

A second thrust area has involved the preparation of thin films of aluminum nitride and the cuprate superconductors by solution deposition methods using polymeric precursors. In the case of AlN, we have investigated the interaction of triethyl- and trimethylaluminum with ethylenediamine and have obtained evidence for several novel intermediates including polymeric derivatives which can be used to make fibers and thin films. Pyrolysis of these films under ammonia was found to give thin, apparently dense, AlN films on Si and other substrates. Our preparation of

the cuprate superconductor films has employed the citric acid/- ethyleneglycol system as a source of both the  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (1-2-3) and Bi/Sr/Cu/O superconductors via solution deposition followed by pyrolysis. Efforts here have focused on the evolution of the cuprate superconductor phases from the polymeric precursor system and have involved the use of IR, TGA, XRD and other methods to follow the conversion process. Successful deposition of "1-2-3" films on  $\text{ZrO}_2$  and  $\text{Al}_2\text{O}_3$  substrates was demonstrated, by several successive dip-coatings with the precursor solution followed, in each case, by pyrolysis to 500 °C in air with a final anneal in oxygen at 950 °C. The intermediate formation of an intimate mixture of  $\text{Y}_2\text{O}_3$ , CuO and  $\text{BaCO}_3$  on pyrolysis of the "1-2-3" precursor system, followed by conversion to the "1-2-3" orthorhombic phase at ca. 850 °C, was evidenced by the XRD data.

The results of these studies have been summarized in twelve papers published or submitted for publication and numerous presentations at scientific meetings and universities. A list of these papers and the meeting presentations follows.

#### Papers Published or Submitted for Publication under the Contract

1. L.V. Interrante, L.E. Carpenter, C. Whitmarsh, W. Lee, G.A. Slack, and M. Garbauskas, "Studies of Organometallic Precursors to Aluminum Nitride", *Mats. Res. Soc. Symp. Proc.* 73, 359 (1986).
2. L.V. Interrante, W. Lee, M. McConnell, N. Lewis, and E. Hall, "Preparation and Properties of Aluminum Nitride Films Using an Organometallic Precursor", *J. Electrochem. Soc.* 136, 472 (1989).
3. L.V. Interrante, Z. Jiang, and D.J. Larkin, "Preparation of the High-Tc Superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  Using Organometallic Precursors", *ACS Symposium Series* 377, 169 (1988).
4. L.V. Interrante, C. Czekaj, and W. Lee, "CVD of AlN and SiC Thin Films Using Designed Organometallic Precursors", NATO ASI Proceedings, "Mechanisms of Reactions of Organometallic Compounds with Surfaces", D.J. Cole-Hamilton and J.O. Williams, eds., Plenum Publ. Corp. (1989) p. 205.
5. W. Lee, L.V. Interrante, C. Czekaj, J. Hudson, K. Lenz, and B-X. Sun, "Chemical Vapor Deposition of Silicon Carbide Using A Novel Organometallic Precursor", *Mats. Res. Soc. Sympos. Proc.* 131, 431 (1989).

6. K. Lenz, K. Rajan, J. Hudson, W. Lee and L.V. Interrante, "Decomposition Kinetics and Film Formation of An Organometallic Precursor to SiC on Si(100)", submitted to *J. Vac. Sci. Technol.*
7. C. Amato, J. Hudson and L.V. Interrante, "Gas Phase Decomposition of an Organometallic Chemical Vapor Deposition Precursor to AlN:  $[(CH_3)_2AlNH_2]$ ", *Mats. Res. Soc. Sympos. Proc.*, Vol. 168, 119-124 (1990).
8. M.M. Lynam, L.V. Interrante, C.H. Patterson, and R.P. Messmer, "Comparison of Isoelectronic Aluminum-Nitrogen and Silicon-Carbon Double Bonds Using Valence Bond Methods", *Inorg. Chem.*, in press.
9. F.C. Sauls, L.V. Interrante, and Z. Jiang, " $Me_3AlNH_3$  Formation and Pyrolytic Methane Loss; Thermodynamics, Kinetics and Mechanism", *Inorg. Chem.*, 29, 2989-96 (1990).
10. Z. Jiang and L.V. Interrante, "1,4-Bistriethylaluminum Ethylenediamine and 1,4-Bistrimethylaluminum Ethylenediamine-Derived Organometallic Precursors to Aluminum Nitride: Syntheses, Structures and Pyrolyses", *Chemistry of Materials*, 2, 439-446 (1990).
11. L.V. Interrante, B. Han, J.B. Hudson and C. Whitmarsh, "Design and Synthesis of CVD Precursors to Thin Film Ceramic Materials", *Applied Surface Science*, (1990), 46, 5-8.
12. Z. Jiang, L.V. Interrante, D. Kwon, F.S. Tham, and R. Kullnig, "Synthesis, Structure, and Pyrolysis of Organoaluminum Amides Derived from the Reactions of Trialkylaluminum Compounds with Ethylenediamine in a 3:2 Ratio", *Inorganic Chemistry*, 30, 995-1000 (1991).

#### Books Edited

"Chemical Perspectives of Microelectronic Materials II", L.V. Interrante, K.F. Jensen, L.H. Dubois and M.E. Gross, eds., *Mats. Res. Soc. Sympos. Proc.*, Vol. 204, (1991) 584 pages. (Symposium E. held at MRS Meeting, Boston, MA, Nov. 1990).



Presentations at Scientific/Technical Conferences (all invited, except as noted)

1. L.E. Carpenter, contributed paper, "Studies of Organometallic Precursors to Aluminum Nitride", Amer. Chem. Soc. Mtg., New York, April 15, 1986.
- 2,3. L.V. Interrante, contributed papers, "Studies of Organometallic Precursors to Aluminum Nitride", MRS Meeting, Palo Alto, CA, April 17, 1986; Electrochemical Society Meeting, Boston, MA, May 6, 1986.
4. "Synthesis and Conversion of Organometallic Precursors to Non-Oxide Ceramics", GRC on Solid State Chemistry, Plymouth, NH, July 28, 1986.
5. "Coordination Compounds as Precursors to Solid State Inorganic Materials", Microsymposium on "Solid State Coordination Chemistry", L.V. Interrante, Chairman, XXIV International Conference on Coordination Chemistry, Athens, Greece, August 27, 1986.
6. "Design and Synthesis of Organometallic Precursors to Refractory Ceramics", Florida Advanced Materials Conference, Palm Coast, FL, December 3-5, 1986.
7. "Organometallic Precursors to AlN and AlN-Containing Ceramics", Workshop on Organometallic Polymers, Argonne Natl. Lab., Argonne, IL, June 1, 1987.
8. "Design and Synthesis of Organometallic Precursors to Ceramic Materials", NSF Organometallic Workshop, Asilomar, CA, June 10, 1987.
9. "Chemical Vapor Deposition of Inorganic Thin Films Using Organometallic Precursors", Symposium on "Chemistry of Microstructures and Electronic Devices", 194th National ACS Meeting, New Orleans, September 4, 1987.
10. "Studies of Organoaluminum Amides as Precursors for the Chemical Vapor Deposition of Aluminum Nitride", Workshop on Group 3-5 Chemistry and Semiconductors, Grand Island, NY, June 4, 1988.
11. "Application of Coordination Chemistry to the Preparation of the  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  Superconductor", Symposium on New Electronic Materials: Synthesis and Characterization", 195th National ACS Meeting, Toronto, June 8, 1988.
12. "CVD of SiC and AlN Thin Films Using Designed Organometallic Precursors", NATO Workshop on "Mechanisms of Reaction of Organometallic Compounds with Surfaces", St. Andrews, Scotland, June 23, 1988.
13. "Application of Organometallic Compounds as Precursors to Solid State Inorganic Materials", GRC on Organometallic Chemistry, Salve Regina College, Newport, RI, June 29, 1988.

14. "Synthesis of Novel Inorganic Materials Using Organometallic Precursors", Gordon Research Conference on Inorganic Chemistry, Brewster Academy, NH, August 3, 1988.
15. "Cyclic  $(AlN)_n$  Compounds as Precursors to Aluminum Nitride", Fifth International Symposium on Inorganic Ring Compounds, Amherst, MA, August 11, 1988.
16. "Molecular Precursor Routes to Electronic Substrates and Thin Films", Symposium on "Molecular Assemblies for Electronics", 196th National ACS Meeting, Los Angeles, CA, September 26-29, 1988.
- 17, 18. "Chemical Routes to Cuprate Superconductor Thin Films", Symposium on "Physical Chemistry of High Temperature Superconductors", 196th National ACS Meeting, Los Angeles, CA, September 26-29, 1988; Symposium on "Frontiers in Materials: Advanced Ceramics", Alfred University, October 21, 1988.
19. "Organometallic Precursors to Ceramic Materials", NY Academy of Sciences, NY, NY, January 12, 1989;
- 20, 21. contributed papers, "Chemical Vapor Deposition of Silicon Carbide Using A Novel Organometallic Precursor", Symposium on "Chemical Perspectives of Microelectronic Materials", MRS Meeting, Boston, MA, Dec. 2, 1988; Fifth Annual Northeast Regional TMS (The Minerals, Metals & Materials Society) Meeting on "Protective Coatings: Processing and Characterization", Stevens Institute of Technology, Hoboken, NJ, May 4, 1989.
22. "Conversion of Organoaluminum Compounds to Aluminum Nitride", The Chemistry of New Materials Conference, SUNY Oneonta, April 22, 1989.
23. "Organometallic Precursors to Ceramic Materials", Florida Advanced Materials Conference, Cocoa Beach, FL, June 12, 1989.
24. "Studies of Coordination Compound Precursors to the Cuprate Superconductors", invited poster and oral presentation, Minisymposium on High Temperature Superconductors, XXVII International Conference on Coordination Chemistry, Brisbane, Australia, July 5, 1989.
25. "The Design and Synthesis of Organometallic Precursors to Ceramic Materials", plenary lecture, Conference on "Applied Organometallic Chemistry", Pokolbin, NSW, Australia, July, 10, 1989.
26. "Studies of Organometallic Precursors to Aluminum Nitride", Symposium on "Chemical Precursors to Ceramics", Inorganic Division, ACS, 198th National Meeting of the Amer. Chem. Soc., Miami Beach, FL, September 12,

1989.

27. "Design and Synthesis of CVD Precursors to Thin Film Ceramic Materials", 2nd Annual "Dielectrics and CVD Metallization Symposium, sponsored by the J.C. Schumaker Co., La Jolla, CA, October 3, 1989.
28. C. Amato, contributed paper, "Mechanistic Studies of the Chemical Vapor Deposition of AlN Films Using Organometallic Precursors", MRS Meeting, Boston, MA, November 29, 1989.
29. L.V. Interrante, "Synthesis of Nonmolecular Inorganic Solids by Pyrolysis of Molecular Precursors", Symposium on "Strategies in Inorganic Synthesis. The First 50 Years", Inorganic Divis., International Chemical Congress of Pacific Basin Societies, Honolulu, Hawaii, December 20, 1989.
30. contributed paper, "Design and Synthesis of CVD Precursors to Thin Film Ceramic Materials", Symposium on "Surface Processing and Laser Assisted Chemistry", E-MRS Meeting, Strasbourg, France, May 29, 1990.
31. "Design and Synthesis of CVD Precursors to Thin Film Ceramic Materials. Symposium on Mechanisms in Chemical Vapor Deposition, Division of Colloid and Surface Chemistry, 200th National Amer. Chem. Soc. Mtg., Washington, D.C., August 27, 1990.
32. "The Conversion of Organometallics to Ceramics - An Emerging Theme in Materials Chemistry", Symposium on "Chemistry in the Age of Materials", Northwestern University, October 30, 1990.

#### Chairing of Meetings

1. Co-chaired and organized session of the Symposium, "Strategies in Inorganic Syntheses. The First 50 Years", Inorganic Div. ACS, International Chemical Congress of Pacific Basin Societies, Honolulu, Hawaii, December 20, 1989.
2. Co-chaired and organized symposium on, "Chemical Perspectives of Microelectronic Materials II", with K.F. Jensen, L.H. Dubois and M.E. Gross, eds., Mats. Res. Soc. Symposium E, held at MRS Meeting, Boston, MA, Nov. 1990.

#### Graduate Students Supported Under the Contract

Wei Lee (M.S. 1988)

Zhiping Jiang (Ph.D. December 1990, currently at MIT Ceramics Res. Lab)

Donna Narsavage (Ph.D. expected, June 1991)

David Larkin (Ph.D. expected, June 1991)

## APPENDIX A

The construction and testing of a cold-wall, hot-stage LPCVD reactor was completed during the period of this research contract. The apparatus consists of a stainless steel chamber containing a 2" diameter electrically heated hot stage interfaced with a precursor vapor source and a vacuum system. In experiments involving the use of single crystal Si as a substrate, the hot stage was replaced by Cu wire leads (terminating in W and Ta clips) for direct resistive heating of the substrate. The Cu leads were connected to an external DC current limited power supply.

The vacuum system includes a turbomolecular pump backed by a two-stage, rotary vane mechanical pump. The operating pressure in the reactor is controlled by an electronically operated butterfly valve between the pumping system and the reactor chamber. A capacitance manometer senses the reactor pressure and, when required, automatically controls the butterfly valve to maintain a constant system pressure. A system of electropneumatic actuated valves and pressure feedback loops provide protection for the turbopump and operator in the event of power failure or operator error.

This apparatus has been interfaced recently with a 200 amu quadrupole mass spectrometer (Hyden Analytical) and a mass flow controller, permitting accurate control of precursor admission rates and on-line analysis of the volatile gaseous products. A diagram which illustrates the main components of this CVD apparatus and their configuration follows.

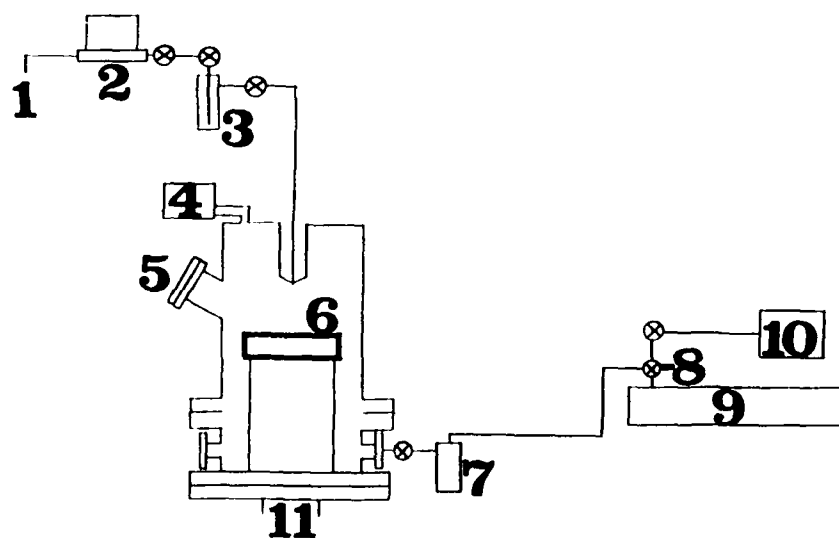


Figure        A schematic of the LPCVD system with an attached QMS; 1.) Carrier gas inlet, 2.) Mass flow controller, 3.) Stainless bubbler containing precursor, 4.) Capacitance manometer, 5.) Sapphire view port, 6.) Silicon substrates, 7.) Series of 3 cold traps, 8.) Sampling leak valve, 9.) QMS, 10.) Mechanical pump used to maintain the gas sample flow, 11.) to the LPCVD main L-N<sub>2</sub> cooled trap and pumping system.